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Publisher's version / Version de l'éditeur:

Powder Technology, 83, 3, 1995

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A hydrodynamic simulation of mineral flotation. Part II: surface chemical effects for coal–oil agglomerate flotation[☆]

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Received 14 February 1994; revised 12 December 1994

Abstract

Oil-agglomeration prior to flotation has proven to be a very effective means of waste fine coal recovery. Existing data on the surface properties of coal–oil agglomerates were used as input parameters for a turbulent, three-phase, computational fluid dynamics simulation of a flotation cell. The flow field in the cell was resolved in two dimensions and the local hydrodynamic conditions were used as the basis for estimating bubble–particle collisions leading to particle collection and flotation recovery. The particulate surface tension as well as a quantity tied to the intrinsic material composition of each coal sample were used as parameters in deriving an expression for the flotation collection efficiency. This integrated simulation, taking into account both local hydrodynamic effects and the surface properties of the particulates was able to predict correctly flotation recovery trends for agglomerates of various coals across a range of oil levels.

Keywords: Flotation; Multi-phase CFD; Coal–oil agglomerates; Surface free energy

1. Introduction

Agglomeration processes bring together fine powders into larger masses in order to improve powder properties. In conventional coal mining, much of the fine coal (typically below 100 mesh) is rejected with tailings as it is impractical to recover. Owing to the heterogeneous nature of coal, smaller particles tend to be either individually richer in ash or in carbonaceous matter compared to the overall composition of the coal. This discretization facilitates effective separation. The tailings are handled in the form of a water slurry and in an agglomeration/separation treatment, an immiscible oil phase is added under high-shear mixing conditions to enhance surface properties. The oil selectively adheres to the carbonaceous surfaces and also acts as a bridging liquid to consolidate several oiled coal particles into an enlarged agglomerate. The ash is excluded and is separated via screening or by selective bubble flotation.

Oil agglomeration combined with bubble flotation has proven to be an effective means of upgrading and recovering waste fine coal when low-oil floc-type agglomerates are formed. Low-oil recovery is desirable, since less ash is incorporated into the agglomerates

and the expense of using oil is reduced. In this state, the flocs are too small and weak for screen separation but can be successfully recovered by flotation as even a small amount of oil imparts a much improved surface character, and agglomerate strength is less important [1–3].

Previous work has provided us with solid surface free energies of coal–oil agglomerates made from coals of different rank, with oil levels varying from 0 to 10 wt.% [4]. It has been illustrated in pilot studies that oil-agglomeration together with flotation gives an enhanced combustibles recovery and ash separation [5,6].

The objectives of this project were multi-fold. It was desired to model the flotation process in a macroscopic fashion via a computational fluid dynamics (CFD) approach. This simulation would be carried out in two dimensions, and would simultaneously treat liquid, gas and solid phases. Further, the flow and turbulence conditions determined for the three-phase flow field would then be used to incorporate a turbulent collision theory into constructing a mechanistically-based model for the recovery of coal–oil agglomerates by flotation. Hydrodynamic forces thus determined could be used to assess the relative contribution of surface forces which are especially important for coal–oil agglomerate flotation, and to account for observed collection effi-

[☆] NRCC No. 37583.

ciencies in a mathematical model. As pointed out in section 4 of part I of this paper, the integration of the particle capture phenomena with the prevailing hydrodynamics will result in different circulation patterns in the top of the cell which depend on the particle diameter. Previous flotation models, such as the ones by Bisshop and White [7] or Kirjavainen [8] have treated the flotation recovery as a unidirectional migration of particles with different rates through the bulk and froth regions.

It is beyond the scope of the present simulation to account for the influence of ash material in the feed, as well as the transport of solids through the froth phase. For these reasons the very low ash Devco coal sample was used in the model for decomposing the hydrodynamic and surface chemical contributions to flotation. The present paper is a first step in incorporating solutions of hydrodynamic field equations for the multi-phase system which occurs in flotation.

The methodology outlined here represents a more integrated approach to flotation modeling in that the micro- events of collision and attachment will be integrated over the flow domain to give a net output.

2. Experimental

The agglomerates were formed from five different coal samples with *n*-hexadecane in a stirred baffled flask under vacuum to eliminate air. Agglomerates of each coal were prepared at oil-levels of 0, 1, 5 and 10 wt.%. They were characterized as described in the following sections.

2.1. Coal analysis

Some of the samples arrived in very large pieces and had to be milled and wet attrition ground, to approximate the state of particulates in waste fines treatment. Details are given in [4]. The particle size distributions were then measured with a Malvern 2600 laser-diffraction instrument. The mean particle sizes of the unagglomerated coals are given in Fig. 1. As one would expect, the mean agglomerate size increases as more oil is added to the system. Fig. 1 is a plot of an enlargement factor F , (the ratio of agglomerate diameter to dispersed particle diameter) versus agglomerate oil level. The enlargement of the lignite samples was slight compared to the higher rank samples. Several measurements (10 or more) were made with each agglomerate sample and the standard deviation of measured means was always less than 1 μm .

The as-received properties of the samples are summarized in Table 1. The coal composition analysis was done on a Leco Mac 400 instrument.

Fig. 1. Enlargement factor vs. oil level for all samples (for hexadecane bridging liquid).

A ratio of the mass fraction of the components that contribute to hydrophobicity (carbon and volatiles) to the hydrophilic components (moisture and ash), is defined as h , the hydrophobicity index, also given in Table 1.

2.2. Surface free energy

The adhesion technique developed by Absolom et al. [9,10] was used to characterize the surface properties of the coal-oil agglomerates. The method is based on the ability of a suspended particle to displace the liquid surrounding it and to form an interface with a solid substrate. The free energy of adhesion, ΔF^{adh} , is expressed as,

$$\Delta F^{\text{adh}} = \gamma_{\text{PS}} - \gamma_{\text{PL}} - \gamma_{\text{SL}} \quad (1)$$

where γ_{PS} , γ_{PL} and γ_{SL} are respectively the particle-substrate, particle-liquid and substrate-liquid interfacial free energies.

Briefly, to determine the surface free energy of a particulate solid, the material is suspended in different cells containing liquids of various surface free energies, with a number of solid substrates onto which the adhesion occurs. Details of this procedure are given by Darcovich et al. [11]. Based on an analysis of the energetics involved [12], when $\gamma_{\text{LV}} \approx \gamma_{\text{PV}}$, neither the particle nor the liquid will be energetically favoured to form an interface with the substrate and the extent of adhesion should be independent of γ_{SV} .

Table 1

Analysis of coal samples [as-received]. Moisture was calculated as a weight percent, on a dry basis

Sample	Volatile material (%)	Fixed carbon (%)	Moisture (%)	Ash (%)	Classification	Sulphur (%)	<di>iameter> (μm)	B.T.U./lb.	h
Devco	40.57	57.34	3.23	2.09	bituminous	0.101	33.0	10102	18.40
Highvale	34.85	44.61	10.74	20.54	subbituminous	0.332	9.4	8171	2.54
Montgomery	44.17	47.26	13.30	8.57	subbituminous	0.624	20.3	9731	4.18
Bien Fait	46.69	38.59	30.57	14.72	lignite	0.584	6.0	9726	1.88
Costello	51.08	39.79	36.32	9.13	lignite	0.823	7.4	10299	2.00

The surface properties of the agglomerates across the range of oil levels tested are shown in Fig. 2. The surface free energies of the unoiled coals fall in the range of 54.0 to 64.0 erg/cm², in agreement with the previous findings of Varga-Butler et al. [13] and Sablik [14]. At 10 wt.% oil, the surface free energies of the bituminous and subbituminous coals closely approximate that of pure hexadecane. The two lignite samples show a more gradual wetting curve, and a surface, which even at 10 wt.% oil, does not exhibit a hexadecane character.

2.3. Flotation tests

For the flotation tests, a Denver laboratory flotation cell was used. Experimental details are given in [4].

The agglomerate flotation recovery data are shown in Fig. 3. Two oils were used in this work, hexadecane and No. 4 fuel oil. The lignite samples simply did not agglomerate sufficiently to be recovered with hexadecane, but some recovery, illustrative of their wettability, was achieved with the heavier, more viscous, No. 4 fuel oil. As reported by Capes and Germain [15], more complex oils contain some inorganic polar species such

Fig. 3. Recovery vs. wt.% oil.

as nitrogen, oxygen and sulphur. These functional groups impart a greater oil wettability to a lower rank, more hydrophilic coal surface such as lignite.

2.4. Numerical flotation simulation

A two-dimensional turbulent flow simulation with mass transfer between two dispersed phases was developed for this work. It was based on existing TURCOM code [16,17]. Full details of its formulation and implementation are given in Part I [19] of this paper.

2.5. Simulation results

A data set for the study of the flotation parameters was constructed by running the three-phase simulation for a combination of particle diameters (corresponding to the agglomerate sizes of the Devco coal-oil agglomerates) and an attachment efficiency, r_a . This data set was then used to deduce a relation between the prop-

Fig. 2. Agglomerate surface tension vs. wt.% oil.

Fig. 4. Recovery vs. d_p from CFD simulation.

erties of the floated material and its recovery. Graphical examples of a typical flow field solution are given in Part I of this paper.

A flotation recovery was calculated for the cell based on the fraction of the solid phase that had been transferred to the gas phase and then exited via the open top outlet. The recovery of the Devco sample plotted against the agglomerate diameter with r_α as a parameter is shown in Fig. 4.

3. Discussion

3.1. Flotation simulation results

The results from the CFD simulation for recovery were plotted in Fig. 4. With r_α as the parameter, the recovery is seen to follow curves containing a maximum recovery at each r_α , and with the maximum becoming less distinct as r_α increases. Dobby and Finch developed a hydrodynamic based micro-flotation model to predict a parameter E_K , which corresponds to the overall probability of flotation P_f [20]. E_K comes from calculating sliding times for particles over bubbles in terms of a collision position model and assigning the induction time parameter to assess the probability of collection.

The data from Fig. 4 and the plot of Dobby and Finch of collection efficiency versus particle size with induction time as a parameter, show a similar functional form. The maximum rate constant is observed at a particle diameter of about 25–30 μm for the model by Dobby and Finch with $d_B = 1.0$ mm. The case for $r_\alpha = 0.2$

(Fig. 5) where recoveries with a range of bubble sizes are predicted with MD-PHASE simulation shows a recovery maximum in a particle diameter range in excellent agreement. For small bubbles (1.0 mm and 0.5 mm), Fig. 5 shows a plateau in recoveries at particle diameters above 25 μm , which corresponds to induction times of less than 20 ms in the Dobby and Finch model. The induction time parameter from the Dobby and Finch model corresponds inversely to the parameter r_α . With an induction time of 0, it means that any contact at all between a particle and a bubble will result in particle collection. The induction time can be considered to be a measure of the surface property of the particle. With reference to coal–oil agglomerate flotation, the oil level used in making the agglomerates improves the solid phase surface for flotation and has a direct bearing on these parameters.

Consider Fig. 5 and the figure by Dobby and Finch where the effect of the bubble diameter is investigated. Again, a similar functional form is observed for both results. At smaller bubble sizes the curves both show a less distinct maximum. Also, results from Dobby and Finch show that the maximum recovery occurs at increasing particle sizes as the bubble diameter is decreased. The CFD simulation results make the same prediction.

From the analysis by Dobby and Finch, the improved E_K with smaller bubbles was explained by saying that despite the smaller surface over which to achieve a particle sliding time, the reduction in gas velocity owing to a lesser bubble diameter still gives an overall increased

Fig. 5. Recovery vs. d_p with d_B as a parameter from CFD simulation.

sliding time. From a macro-hydrodynamic point of view smaller bubbles are more subject to the influence of the liquid and solid phase flow fields since there is a less strong buoyancy force driving them upwards. Hence, their streamlines are less direct between the inlet and the outlet, allowing for a higher residence time and better mixing of the three phases in the turbulence, and hence more frequent collisions. Further, at a constant air flux, smaller bubbles will produce a higher number density, and thus again, more collisions.

It is interesting to note that the approach of Bischofberger and Schubert [18] which is based on collisions arising through turbulent energy dissipation, and which was incorporated in the present CFD model, gives quite similar trends in collection and recovery as a function of the particle diameter as does the model of Dobby and Finch. This indicates that the local hydrodynamic forces under the prevailing turbulence in a many-particle float cell produce collisional estimates of a nature similar to those from the laminar two-body approach resistance method, used in the model of Dobby and Finch, when individual particle interactions are scaled up to a unit operation. The parallel results thus suggest that the micro-flotation interactions of an essentially laminar nature may be quite valid within length scales less than the turbulent mixing length.

3.2. Agglomerate surface property flotation model

An objective was to correlate the observed flotation recovery with the surface free energy of the agglomerates. The parameter for flotation recovery which directly depends on the surface properties is the attachment efficiency, r_a . The CFD model is intended to predict a recovery, given the flow conditions, particle and bubble diameters and a value for r_a which should depend on the measured agglomerate surface free energy.

The model in this case can be developed as a monotonic relation between the attachment efficiency, r_a , and the agglomerate surface tension due to oil wetting γ . The effect of the bubble diameter would be absorbed into model constants, since it was kept constant in the numerical simulations and the laboratory float cell tests. For expressing r_a as a function of γ , the agglomerate diameter was not included as it could not be decoupled from the oil level and as such, inherently had its contribution input.

The Devco sample was used as a calibrating sample simply because there were six data points available for all the required parameters, while there were only three data points for the other coals. The data in Fig. 4 were numerically interpolated to give values of r_a which correspond to the measured flotation recoveries of the Devco sample at each oil level. These values of r_a are plotted versus corresponding γ in Fig. 6 and are indicated

Fig. 6. Calculated r_a vs. γ for Devco sample.

by the squares. In order to obtain a simple relation that behaves according to the data in Fig. 6, a hyperbolic functionality is adopted [21]. That is,

$$\frac{1}{r_a} = m\gamma + b \quad (2)$$

This model contains two asymptotes; one at $r_a = 0$ with a physical meaning of zero recovery for infinitely hydrophilic particles, and the other at $\gamma = -b/m$ for a full oil surface and maximum bubble-particle attachment. The regression analysis was done with Eq. (2) and the regression curve is also shown in Fig. 6. The parameters b and m are simply the regression coefficients. The abscissa asymptote is calculated as 23.5 erg/cm² while the actual surface tension of hexadecane is 27.6 erg/cm². The scope of this correlation should be limited to the present oil level range since above it, a regime will exist of constant coal-oil agglomerate surface character, but with still increasing agglomerate diameters. Further, if Eq. (2) is employed within the oil level range used in the experiments, then the calculated values for r_a are plausible and stay well within the physically realizable limits of 0 and 1.

An analysis by Al Taweel et al. [22] gives the overall probability of flotation P_f , as a product of various subprocesses.

$$P_f \propto P_c P_a (1 - P_d) \quad (3)$$

Above, the subscripts c, a and d refer respectively to collision, attachment and detachment. The collision

part was dealt with hydrodynamically which leaves the terms P_a and P_d for consideration. In this project, there was no real means of decoupling P_a and P_d , but their functionality can be formulated, based on the above considerations by Al Taweel et al. [22], as,

$$P_a(1-P_d) \propto \frac{f_1(\text{surface properties})}{f_2(\text{particle diameter})} \quad (4)$$

To extend the model to coal–oil agglomerates in general, the agglomerate diameter d_p , is considered, and from Eq. (4), it must be inserted into the model such that when the collision is *already* assumed to have occurred, a smaller particle (agglomerate) will more readily attach to a bubble. Further, it is known that coals of a higher rank are better recovered. To account for this, we have the hydrophobicity index, h which was determined as a function of oil level from deashing data from [4].

With reference to the Devco sample as a calibration sample, the model of Eq. (2) can be generalized by the empirical factor, $(d_{PCAL}/d_p)^{\beta h}$, where d_{PCAL} is the diameter of an agglomerate of the Devco coal. By running this CFD simulation at a range of values for the exponent, a value of $\beta=0.5$ was found to give the best fit.

So, for a more general model to calculate a value of r_a from the coal–oil agglomerate properties, which can then be input into the CFD simulation to predict flotation recoveries, we have,

$$r_a = \left(\frac{d_{PCAL}}{d_p} \right)^{h/2} / (m\gamma + b) \quad (5)$$

Table 2 lists the attachment efficiencies associated with each type of agglomerate to be input to the CFD simulation. These simulations were run, and the calculated recoveries are shown with the dashed lines in Fig. 7, compared to the measured recoveries. The predicted values appear to follow the measured data in Fig. 7 quite well. It should be noted that the results for the Highvale coal show an increasing recovery as the oil level is increased, while the Montgomery samples passes through a recovery maximum at an intermediate

Table 2

Parameter inputs for flotation simulation for subbituminous coals for recovery predictions

Sample	Oil (wt.%)	γ	$\frac{d_{PCAL}}{d_p}$	h	r_a
Highvale	1.0	31.66	3.63	6.05	0.1466
Highvale	5.0	29.93	3.96	6.03	0.1625
Highvale	10.0	26.53	3.78	6.01	0.1890
Montgomery	1.0	32.73	1.59	6.64	0.0714
Montgomery	5.0	28.42	1.63	6.64	0.0925
Montgomery	10.0	30.40	1.54	6.62	0.0749

Fig. 7. Integrated model recovery predictions.

oil level. This is an effect of the absolute size of each sample. The Highvale coal ranges from 9.4 to 14.1 μm , falling into a size range in Fig. 4 where the recovery increases monotonically with agglomerate diameter. The Montgomery sample, in contrast, has a size range of 20.3 to 34.5 μm , which passes through the recovery maximum in terms of agglomerate diameter shown in Fig. 4. Thus, the model for attachment efficiency, integrated with CFD simulation, is able to account for these interdependent effects when calculating a recovery.

3.3. A discussion of flotation kinetics modeling

There are several known trends and phenomena in flotation and it is the purpose of this section to discuss the results and observations from the present study in view of the overall body of knowledge available in the field.

In general, the influence of an increased bubble size is to reduce the recovery by flotation. This has been observed by Anfruns and Kitchener [23] as well as by Weber [24], and was incorporated into a ratio of d_p/d_B by Reay and Ratcliffe [25]. This is consistent with the findings of this work. This result would be manifested by the reduced residence time in the cell due to buoyancy forces, and by being less subject to the turbulent mixing which determines the collision rate according to the equation of Bischofberger and Schubert [18] as discussed in Part I of this series [19].

The effects of the particle size likewise are often put into expressions for the flotation kinetics. Kinetics are typically applied to a batch system, but the rate of flotation given for such a system directly relates to the collection efficiency in a continuous system. Reay and Ratcliffe [25] comment that the flotation rate was

found for particles up to 15 μm proportional to,

$$R_t = k_t \left(\frac{d_p}{d_B} \right)^{2.05}$$

At particle sizes larger than 15 μm , the attachment efficiency was said to become very dependent on d_p , and the exponent was found to become even greater than 2.05. In such a specialized study, the hydrodynamic influence of larger particles could not be accounted for. The present study can easily treat bubble and particle size effects independently. Certain hydrodynamic assumptions were made by Reay and Ratcliffe which they considered valid for $d_p/d_B < 0.25$. The present work makes no such assumptions. Note that the Reay and Ratcliffe model is for the collision efficiency only. The exponential term from Eq. (5) is for the subsequent mechanism of attachment where the particle diameter effect is opposite than for collision.

A more updated model, based on some of the original work by Reay and Ratcliffe was presented by Dobby and Finch [20]. Independent effects of bubble and particle size were given by them. However, the model goes only as far as predicting values of a collection efficiency E_K , and how this parameter is manifested in a three-phase float cell is left unresolved. The work presented here integrates the collision models and flow field effects and can treat ranges of the input parameters as prescribed by the local conditions in the float cell. That is, variables such as dispersed phase concentrations, velocities and bubble loading are treated in an interdependent and dynamic way, so that these effects need not be generalized or shown one at a time in an illustrative sense.

The influence of the rank of a coal and hence its flotability is often expressed in terms of an induction time. This factor reduces to a probability function arising in a statistical cell model for collision frequencies. If a field solution is used to account for the collisions, as done in this work, this probability which is determined as an integral over all possible collision angles, can be replaced in the model with a quantity tied to the surface properties. In the absence of surface free energy data, an induction time which is compared to an averaged, integrated sliding time is used as the attachment efficiency. A value of the agglomerate surface free energy uniquely corresponds to this integrated probability.

In this work, the exponent used was $h/2$ since it was a quantity relating the flotability of the coal to its material composition. Thus, this work has provided a measured quantity reflective of the particulate material character, used to regulate the determination of r_a values. A paper by Fuerstenau et al. [26] discusses the flotability in terms of surface functional groups in coals. The content of phenolic and carboxylic groups correlated very strongly with the flotation response. Coal wettability

was found to depend on the balance of hydrophobic and hydrophilic sites, and in an analogous fashion, the attachment efficiency equation developed here employed the agglomerate surface tension and hydrophobicity index.

The present approach may appear to be excessively complex as it may seem that simply making a least squares correlation between the particle surface tension and flotation recovery would illustrate the phenomenon. Such a method could not account for the simultaneous agglomerate size enlargement which occurs as the oil level is increased to improve the surface properties. The agglomerate size impacts on both the collision and attachment mechanisms, the macroscopic hydrodynamic behaviour, as well as the number density of solids in the flotation cell at a constant solids loading. The present approach considers all these simultaneous, interdependent factors first for estimating a value of r_a and then makes use of the physical properties of the agglomerate in the hydrodynamic simulation. Further, the geometrical simplifications make it such that no claim is made for providing physically exact float cell flow fields, rather a plausible environment is created wherein the mechanisms of flotation which are at work can be decoupled to show the influences of both of the collisional and attachment probabilities. This decoupling of the two probabilities is a novel advance.

4. Conclusions

From the work carried out, it can be said that there is a direct correlation between the measured surface free energy of coal–oil agglomerates in aqueous media and the agglomeration/flotation recovery results. Coals of subbituminous and bituminous rank will acquire an oil-like surface at oil levels as low as 1 wt.%. Lower ranked lignites show a decrease in surface free energy as oil level is increased, but their ability to be agglomerated and floated is limited.

A two-dimensional, three-phase finite-volume CFD simulation including collisions and attachment of the dispersed phases was developed. By altering input values of an attachment efficiency and the particle sizes, the behaviour of the float cell was characterized and correlated to the measured surface properties of the Devco bituminous coal.

An attachment efficiency accounting for agglomerate size, inherent material hydrophobicity and oil wettability was input to the CFD simulation of the float cell, and accurately predicted the recoveries of two other subbituminous coal samples. The integrated flotation model with resolved field equations determining recovery is a novel method.

5. List of symbols

b	regression model constant
d_B	bubble diameter
d_P	particle diameter
E_K	flotation collection efficiency
F	agglomeration enlargement factor
h	hydrophobicity index
k_f	Reay and Ratcliffe model coefficient
m	regression model constant
n	Reay and Ratcliffe model exponent
P_a	probability of attachment
P_c	probability of collision
P_d	probability of detachment
P_f	probability of flotation
r_α	attachment efficiency

Subscripts

CAL calibration sample

Greek letters

β	empirical model exponent coefficient
ΔF^{adh}	free energy of adhesion per unit area
γ	agglomerate surface tension
γ_{LV}	liquid–vapour interfacial tension
γ_{PL}	particle–liquid interfacial tension
γ_{PS}	particle–substrate interfacial tension
γ_{PV}	particle–vapour interfacial tension
γ_{SL}	substrate–liquid interfacial tension
γ_{SV}	substrate–vapour interfacial tension

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